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# **A detailed post-IR IRSL dating study of the Niuyangzigou loess site in northeastern China**

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Yi, S.W., Buylaert, J.-P., Murray A.S., Lu, H.Y. Thiel, C. & Zeng L.: A detailed post-IR  
IRSL dating study of the Niuyangzigou loess site in northeastern China.

In this study, we report standard quartz SAR OSL and post-IR infrared (IR) stimulated  
luminescence (post-IR IRSL; pIRIR<sub>290</sub>) measurements made on sand-sized quartz  
and K-feldspar extracts from the loess-palaeosol sequence at Niuyangzigou in  
northeastern China. The quartz OSL characteristics are satisfactory. Extensive  
pIRIR<sub>50,290</sub> dose recovery tests were performed by adding doses on top of the natural  
dose. We found that dose recovery ratios improve significantly when the test dose  
ranges between ~15 and ~80% of the total dose, and good dose recovery (within  $\pm 5\%$   
of unity) can be obtained up to ~800 Gy. Otherwise, the dose recovery ratio deviates  
from unity. The  $D_e$  values also depend on the test dose size so we conclude that the  
effect of test dose size should be routinely considered in pIRIR dating. First IR  
stimulation plateau pIRIR<sub>290</sub> results are compared with multiple elevated temperature  
-pIRIR (MET-pIRIR) data. It appears that the low temperature MET-pIRIR data are  
strongly affected by poor dose recovery, but this is not the case for the pIRIR<sub>290</sub>

results. Natural signal measurements at the highest (first IR) stimulation temperature on a sample expected to be in field saturation, suggest ~10% signal loss is present in pIRIR signals. Long term laboratory bleaching experiments (>80 days) show that a constant (or very difficult to bleach) residual pIRIR<sub>290</sub> signal is reached after ~300 h, corresponding to a dose of  $6.2 \pm 0.7$  Gy. Quartz OSL and feldspar pIRIR<sub>50,290</sub> ages are in good agreement at least back to ~70 ka. Beyond this the quartz ages begin to underestimate but the feldspar ages are in agreement with the expected Last Interglacial age palaeosol.

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Northeastern China is located in the East Asian monsoon region and lies near the present-day limit of the summer monsoon (Fig 1); as a result it is sensitive to the global climate systems of both the high and low latitudes. Since the 1980s this area

has been characterised by a pronounced regional temperature increase and a decrease in precipitation compared to the rest of China (Sun *et al.* 2007; Gao *et al.* 2008). Because of these factors it is a very suitable place to study climate change and environmental evolution during the late Quaternary. The considerable area of loess deposits in north China forms one of the largest and most important aeolian records on Earth. Loess/palaeosol sequences contain detailed archives of terrestrial palaeoenvironmental changes and are highly sensitive to climatic changes, specifically to shifts in the Asian summer and winter monsoon and/or Northern Hemisphere westerly circulation (Liu & Ding, 1998). However, research into loess deposition and past climate change in northeastern China is limited due to the lack of independent age control (i.e. radiometric dating).

Luminescence dating has proved to be particularly successful for dating aeolian sequences (e.g. Stevens *et al.* 2006; Buylaert *et al.* 2008; Lai 2010; Lai & Fan 2014). These studies are all based on equivalent dose estimation using the single aliquot regenerative (SAR) dose protocol developed for fast-component dominated quartz OSL (Wintle & Murray 2006). However, its use is typically limited to samples with equivalent doses up to 150-200 Gy. This restricts the OSL dating of quartz to loess deposits (typical dose rate of between 3 and 4 Gy ka<sup>-1</sup>) from within the last 50-70 ka (Buylaert *et al.* 2007; Roberts 2008; Chapot *et al.* 2012; Timar-Gabor & Wintle 2013). The infrared stimulated luminescence (IRSL) signal from feldspar (Hütt *et al.* 1988)

65 has the potential to extend the datable age range because it saturates at much higher  
66 doses compared to quartz OSL (Huntley & Lamothe 2001). However, it is now widely  
67 accepted that IRSL measured at ambient temperature suffers from anomalous fading  
68 (e.g. Spooner 1994; Huntley & Lian 2006; Buylaert *et al.* 2011, 2012). Recent  
69 advances in the understanding of feldspar as a luminescence dosimeter (Thomsen *et*  
70 *al.* 2008, 2011; Murray *et al.* 2009; Jain & Ankjærgaard 2011) have led to new  
71 single-aliquot regenerative dose (SAR) dating protocols (so called post-IR IRSL  
72 protocols; e.g. Buylaert *et al.* 2009), in which a high temperature IRSL signal is  
73 measured at an increased temperature after a first IR stimulation at some lower  
74 temperature, usually close to ambient temperature. These post-IR IRSL (pIRIR)  
75 signals appear to be much less prone to fading than the conventional IRSL signal.  
76 Various single-aliquot based pIRIR dating protocols for feldspar have been developed;  
77 these include a two-step (e.g. Thomsen *et al.* 2008; Buylaert *et al.* 2009; Thiel *et al.*  
78 2011a) and a multiple elevated temperature (MET) (Li & Li 2011; 2012a) pIRIR  
79 stimulation procedure. These procedures have been shown to give accurate ages  
80 both for young (<10 ka, Fu & Li 2013; Reimann *et al.* 2011, 2012) and old (>100 ka,  
81 Buylaert *et al.* 2012; Li & Li 2011, 2012a; Kars *et al.* 2012; Zander & Hilgers 2013)  
82 samples. Even though pIRIR measurement protocols have become the preferred  
83 method to measure feldspar equivalent doses, there are still remaining issues  
84 concerning dose recovery results (Roberts 2012), the determination of a potentially  
85 unbleachable component (e.g. Buylaert *et al.* 2011, Stevens *et al.* 2011; Murray *et al.*

2012; Kars *et al.* 2014a) and signal (in)stability (Li & Li 2012b; Thomsen *et al.* 2011).

The present work focuses on the Niuyangzigou (NYZG) loess-palaeosol sequence in northeastern China (Fig. 1). The luminescence characteristics of quartz SAR OSL and K-feldspar pIRIR signals (pIRIR<sub>290</sub>) are documented in a SAR-based methodology. The pIRIR signal measured at 290°C is then investigated in detail to (i) test the dependence of dose recovery and  $D_e$  on test dose size, (ii) determine the size of an (un)bleachable residual component and (iii) check the stability of the signal (including MET-pIRIR signals) using a sample expected to be of non-finite age. Finally, quartz OSL and pIRIR<sub>290</sub> ages are compared and a luminescence chronology for the upper part of the NYZG section is presented.

## **Geological setting, stratigraphy and sampling**

Northeastern China extends from 40° to 59° N and 110 to 135° E, it includes the provinces of Heilongjiang, Jilin and Liaoning, and the Inner Mongolia Autonomous Region east of 110° E (Fig. 1). The major topographical features in this area are the extensive Northeast Plain surrounded by a series of mountains and hills. The Daxingan Mountains lie along the western side while the Xiaoxingan Range with a northwest-southeast orientation lies to the north.

The NYZG section (41°55' N, 118°43' E, 774 m.a.s.l) is situated in KaLaQin County, Chifeng city, in the northeastern part of the Inner Mongolia Autonomous Region (Fig. 1). The whole section is a 36.6 m thick series of loess intercalated by palaeosols; it is accessible through a natural exposure of the upper 26 m and a 10.6 m deep exploratory well. The magnetic characteristics of the Matuyama–Brunhes palaeomagnetic boundary (0.78 Ma; Cande & Kent 1995) have been identified towards the bottom of the sequence and some evidence for the Jaramillo sub-chron is exposed in a basal complex of palaeosols (Zeng *et al.* 2011).

Fifteen luminescence samples were collected using light-tight steel cylinders (diameter 5 cm, length 20 cm) from the upper 3.2 m of a freshly excavated profile. Based on field observations, this section comprises the upper Holocene soil ( $S_0$ ), the Last Glacial loess ( $L_1$ ) and the Last Interglacial palaeosol ( $S_1$ ) (Fig.1). One additional sample (sample code 1535) was collected near the B/M boundary (Zeng *et al.* 2011) to provide a feldspar sample of non-finite luminescence age (expected burial dose >2000 Gy).

## **Sample preparation and analytical facilities**

Samples were opened under subdued red light conditions and material from the outer ends of each tube was used for dose rate and water content measurement. The

128 non-light exposed material from the middle part of the tube was treated with HCl (30%)  
 129 and H<sub>2</sub>O<sub>2</sub> (30%) to remove carbonates and organic matter, respectively. Grains in the  
 130 range 63 to 90 µm were obtained by wet-sieving. Pure quartz grains (no significant  
 131 IRSL signals) were obtained after a 40 min HF (40%) etch and 40 min 10% HCl rinse.  
 132 For K-rich feldspar extraction, a portion of the initial 63-90 µm sieved fraction was  
 133 cleaned with 10% HF for 20 min to remove coatings and the outer alpha irradiated  
 134 layer, and then rinsed in 10% HCl acid for 20 min to remove any precipitated fluorides.  
 135 K-rich feldspars were floated off using an aqueous heavy liquid (sodium  
 136 heteropolytungstate 'LST Fastfloat'; density 2.58 g.cm<sup>-3</sup>).

137

138 Luminescence measurements employed Risø TL/OSL readers model DA-20  
 139 (Bøtter-Jensen *et al.* 2003) equipped with blue LEDs (470 nm, ~80 mWcm<sup>-2</sup>) infrared  
 140 (IR) LEDs (870 nm, ~135 mWcm<sup>-2</sup>); each reader was equipped with a calibrated <sup>90</sup>Sr  
 141 /<sup>90</sup>Y beta source. Quartz OSL signals were collected through 7.5 mm of Schott U-340  
 142 (UV) glass filter and feldspar (post-IR) IRSL through a combination of Corning 7-59  
 143 and Schott BG-39 glass filters (blue-violet part of the spectrum). Quartz grains were  
 144 mounted as large (8 mm) aliquots on stainless steel discs and K-rich feldspars as  
 145 small (2 mm) aliquots on stainless steel cups; Silicone oil (Silkospray) was used as an  
 146 adhesive. For quartz OSL, the signal was derived from the first 0.16 s of stimulation  
 147 and an early background (0.16-0.32 s) to minimize the influence of slow and medium  
 148 components (Ballarini *et al.* 2007; Cunningham & Wallinga 2010). Feldspar (post-IR)



IRSL signals were derived from the integral of the first 2 s of (post-IR) IRSL stimulation, less a background based on the last 50 s; for measurements made using an MET-pIRIR protocol these intervals are 1 and 25 s, respectively.

## Dosimetry

The environmental dose rate was calculated from the uranium, thorium and potassium concentrations, measured by neutron activation analysis (NAA). The *in situ* water content (mass of moisture/dry mass) was determined by weighing the sample before and after drying, and was assigned an absolute uncertainty of  $\pm 5\%$  (e.g. for a water content of 10% we have used  $(10 \pm 5)\%$ ). Using the revised dose rate conversion factors of Guérin *et al.* (2011) and water content attenuation factors (Aitken 1985), the elemental concentrations were converted into effective dose rate. Calculation of the cosmic dose rate is based on Prescott & Hutton (1994). For K-feldspar dose rates a K concentration of  $12.5 \pm 0.5\%$  and Rb concentration of  $400 \pm 100$  ppm was assumed (Huntley & Baril 1997) consistent with earlier work on sand-sized K-feldspar from Chinese loess (Zhao & Li 2005; Li *et al.* 2008). A small internal dose rate contribution from U and Th of  $0.03 \pm 0.015$  Gy ka<sup>-1</sup> and  $0.06 \pm 0.03$  Gy ka<sup>-1</sup> was included for quartz and K-feldspar respectively (Mejdahl 1987; Zhao & Li 2005; Vandenberghe *et al.* 2008). Table 1 summarises the uranium, thorium and

potassium concentrations and the resulting total dose rates to quartz and K-feldspar grains

## **Quartz OSL characteristics and resulting ages**

The quartz equivalent doses ( $D_e$ ) were measured using a standard SAR protocol (Murray & Wintle 2000, 2003; Table 2). Typical dose response curves and OSL decay curves (inset) are shown for the upper (138101) and lower (138115) samples in Fig. 2A and B, respectively. The blue-light stimulated OSL signals decrease very quickly during the first second of stimulation, indicating that the signal is dominated by the fast component (Jain *et al.* 2003; Singarayer & Bailey 2003). In order to select appropriate preheat conditions, the  $D_e$  dependence on preheat temperature was checked using a preheat plateau test. A plateau was observed for temperatures from 180 °C to at least 260 °C (Fig. 2C). The suitability of our adopted SAR protocol was further checked with a dose recovery test (Murray & Wintle 2003). The ratios of the given doses to the measured doses were within 10% of unity over the entire temperature interval (Fig. 2D). Based on these preheat tests, a 260 °C preheat for 10 s and 220 °C cut-heat was chosen for final  $D_e$  determination. For all samples, recuperation is low (average =  $0.06 \pm 0.01\%$  of natural,  $n = 245$ ) and the average recycling ratio is  $1.01 \pm 0.01$  ( $n = 233$ ) indicating that the adopted SAR protocol successfully corrects for laboratory sensitivity changes.

190

191        In order to ensure that only those aliquots that are capable of measuring the  
 192 dose of interest have been included in the calculation of the mean  $D_e$ , aliquots with  
 193  $2 \times D_0$  values that are smaller than the average dose (derived from all aliquots) are not  
 194 used in the derivation of  $D_e$ , irrespective of the  $D_e$  value of the individual aliquot  
 195 (based on Thomsen *et al.* 2016; note that for simplicity our data were approximated  
 196 by a single saturating exponential function). For the upper four samples (<150 Gy) no  
 197 aliquots were rejected based on this criterion. Below this level (1.20 m) the  
 198 percentage of aliquots that are rejected ranges between 6 and 31%. Over all the 15  
 199 samples the average  $D_e$  was decreased by ~3% because of this selection criterion;  
 200 the biggest decrease was 16% and the largest increase was only 2%. Table 1  
 201 summarises the resulting  $D_e$  values and quartz OSL ages. The calculated  $D_e$  values  
 202 range from  $92 \pm 3$  Gy for the upper sample to  $270 \pm 13$  Gy for the lowest sample; the  
 203 corresponding ages are  $30 \pm 2$  and  $99 \pm 7$  ka. We note that the bottom four samples all  
 204 have  $D_e$  values  $\geq 200$  Gy. Several papers suggest that the upper limit of SAR based  
 205 quartz OSL dating lies around ~200 Gy in loess and ages derived from these high  
 206 doses should be interpreted with caution because they are likely to be  
 207 underestimates (e.g. Buylaert *et al.* 2007, 2008; Zhou & Shackleton 2001; Lai 2010;  
 208 Chapot *et al.* 2012, Timar-Gabor & Wintle 2013; Lai & Fan 2014).

209

## 210 **Feldspar luminescence characteristics**

## 211 ***Dose response curve and $L_x/T_x$ plots***

212 The SAR pIRIR<sub>290</sub> protocol proposed by Thiel *et al.* (2011a) and tested by Buylaert *et*  
 213 *al.* (2012) was used to measure the K-feldspar dose in these samples (Table 2).  
 214 Aliquots were preheated at 320 °C for 60 s followed by IR diode stimulation (90%  
 215 power) at 50 °C for 200 s (the choice of this temperature is discussed in the next  
 216 section) to recombine nearby electron-hole pairs (Jain & Ankjærgaard 2011).  
 217 Subsequently, a more stable IRSL signal is measured at 290 °C for 200 s (referred to  
 218 as pIRIR<sub>290</sub>); this is the dating signal of interest. The response to the test dose is  
 219 measured in the same manner and is followed by an IR illumination at 325 °C for 200  
 220 s at the end of each SAR cycle, to reduce recuperation. Representative K-feldspar  
 221 pIRIR<sub>290</sub> and quartz OSL dose response curves (normalised to the fitted saturation  
 222 values) are presented for the lowermost sample (138115) in Fig. 3A. It can be seen  
 223 that the feldspar pIRIR<sub>290</sub> dose response curve has a much more extended dose  
 224 range compared to quartz OSL, 86% of saturation is reached at 1250 and 310 Gy,  
 225 respectively. This extended feldspar range indicates its usefulness for dating samples  
 226 beyond the quartz OSL limit.

227

228 One of the main assumptions when using the SAR protocol to measure a dose is  
 229 that the test dose luminescence sensitivity is directly proportional to the preceding  
 230 regenerative dose; i.e. it can correct for sensitivity changes (Murray & Wintle 2000).  
 231 We have constructed  $L_x$ - $T_x$  plots for the pIRIR<sub>290</sub> signal for five samples down our

section by repeating SAR cycles using a fixed regenerative dose and test dose (Fig. 3B); there is clear proportionality between regenerative and test dose signals indicating that the test dose successfully corrects for sensitivity changes. This is also supported by the mean recycling ratio of  $1.031 \pm 0.002$  ( $n = 109$ ,  $RSD = 2\%$ ; 15 samples). If no test dose is used for sensitivity correction, the resulting recycling ratio is  $1.099 \pm 0.011$  with a much higher standard deviation of 10%. For all samples, recuperation is small ( $<3\%$  of the natural signal) showing that our high temperature clean-out is sufficiently stringent. However, the most stringent test for any SAR protocol is the dose recovery test (Murray 1996; Wallinga *et al.* 2000; Murray & Wintle 2003) and this is addressed in the next section.

### **Dose recovery test**

Although several studies have presented good/acceptable dose recovery results for pIRIR<sub>290</sub> protocols on a variety of sediments (e.g. Buylaert *et al.* 2011, 2012, 2013; Nian *et al.* 2012; Thiel *et al.* 2012; Tsukamoto *et al.* 2014), there is also considerable evidence for poor dose recovery results (e.g. Stevens *et al.* 2011; Lowick *et al.* 2012; Roberts 2012; Thiel *et al.* 2011b, 2014 ; Murray *et al.* 2014). Usually, these poor dose recovery ratios were significantly greater than unity. Some authors report difficulties in bleaching natural pIRIR<sub>290</sub> signals (using SOL2 simulator or natural daylight); these can result in poor dose recovery because of incorrect residual dose estimation (e.g. Stevens *et al.* 2011; Alexanderson & Murray 2012). To avoid potential complications

related to bleaching natural samples, a dose recovery test can also be performed on modern/young samples by adding a beta dose on top of a relatively small natural dose (e.g. Buylaert *et al.* 2011); this is the approach taken in this study.

We investigated the dependence of dose recovery ratios on test dose size by adding beta doses (ranging from 99 to 1593 Gy) to aliquots of the uppermost sample (138101) which has a pIRIR<sub>290</sub> D<sub>e</sub> value of ~100 Gy. The dose recovery ratio was calculated as the measured dose divided by the sum of the natural and the given dose. Qin & Zhou (2012) suggested that the dose recovery ratio is dependent on the test dose size (their Fig. 3B) but their data are limited in test dose range and inconclusive (the data for different test dose sizes does not differ significantly). Fig 4A shows the dose recovery ratio as a function of test dose size over a wide test dose range (5-260% of the total (natural+added) dose). For small test doses (<15%), the dose recovery ratio is significantly greater than unity. In contrast, large test doses (>80%) yield ratios lower than unity. The best dose recovery ratios (within  $\pm 5\%$  of unity) are found when the test dose ranges between ~15 and ~80% of the total dose. The data of Fig. 4A are also shown in Fig. 4B but now as a measured versus added dose plot; these are so-called Single Aliquot Regenerative Added dose (SARA; Mejdahl & Bøtter-Jensen 1994; Wallinga *et al.* 2001) dose response curves. As expected from the dose recovery ratios, the slope ( $1.01 \pm 0.02$ ) of the fitted line is indistinguishable from unity when test doses between 15 and 80% are used. It is

interesting to note that at least for added doses larger than about 600 Gy, the data measured with small (<15%) or large (>80%) test doses do not follow a simple straight line relationship between measured and added dose, rather the measured dose increasingly deviates from the known added dose; this occurs because of a systematic change in  $D_0$  with test dose.

We conclude from the data of Fig. 4 that, at least for these samples, an acceptable dose recovery ratio is best ensured by using a test dose in the range 15-80% of the total (natural + added) dose.

#### ***Equivalent dose***

***Effect of test dose size.*** Fig. 5 shows the dependence of  $D_e$  on test dose size for the uppermost (138101), middle (138108) and lowermost (138115) samples from the section. Sample 138101 shows a clear  $D_e$  plateau for test doses ranging between 5 and 80% of the  $D_e$  value; the  $D_e$  value of the older samples appears insensitive to test dose size over a shorter range, up to only ~60%. The shape of the dose recovery ratio versus test dose curve (Fig. 4A) broadly resembles the shape of the  $D_e$  versus test dose curve but it appears that especially for low test doses (<20%) the  $D_e$  value is less sensitive to test dose size than the dose recovery value. Nevertheless, for these samples, it appears to be inappropriate to use very small (<10%) or very large (>60%) test doses because of poor dose recovery ratios. For the remainder of this study we

adopt a test dose size of ~30% for  $D_e$  measurements; in this range the dose recovery ratio is within  $\pm 5\%$  of unity and the  $D_e$  values are consistent with the plateau regions in  $D_e$  versus test dose graphs.

**Comparison with MET-protocol.** Based on the model prediction by Jain & Ankjærgaard (2011) that the stability of the post-IR IRSL signal could be dependent on the first IR stimulation temperature and/or wavelength, Buylaert *et al.* (2012) have suggested the use of a first IR stimulation temperature plateau to investigate whether a stable signal was reached (interval over which  $D_e$  is insensitive to prior IR stimulation temperature). Fig. 6A,B shows  $D_e$  as a function of prior IR stimulation temperature for the top (138101) and bottom (138115) samples of the section, respectively; from these data it seems that for  $D_e$  values up to ~400 Gy the  $D_e$  is insensitive to first IR stimulation temperature and a stable signal is observed. This is in agreement with the data of Li & Li (2012b) who compared pIRIR<sub>50,290</sub>  $D_e$  values with pIRIR<sub>200,290</sub>  $D_e$  values and MET-pIRIR<sub>250</sub> results for Luochuan samples and showed that the three methods are indistinguishable back to ~400 Gy; they also showed that beyond this a low temperature IR cleaning at 50 °C is apparently not sufficient to recombine all nearby electron-hole pairs. Beyond ~400 Gy, pIRIR<sub>200,290</sub> is consistent with the MET-250 data. It is claimed by Li & Li (2011, 2012a, b) and Fu & Li (2013) that the MET-pIRIR protocols have an advantage over the two-step protocols (Buylaert *et al.* 2009, 2012; Thiel *et al.* 2011a) because of the possibility to construct



316 “age-temperature” plots from individual aliquots; the presence of an “age plateau” at  
317 higher stimulation temperatures is used to determine whether a stable signal was  
318 identified. We have also measured the MET-pIRIR signals (following Li & Li, 2012 a)  
319 for the top and bottom sample of our section and the data are also shown in Fig. 6A,B  
320 (open symbols). As expected the MET-pIRIR  $D_e$  values are consistent with the  
321 pIRIR<sub>290</sub>  $D_e$  measurements when stimulation temperatures of >200 °C are reached. It  
322 should be noted that the plateau identified by Li & Li (2011, 2012a) and Fu & Li (2013)  
323 is usually limited to two (or maximum three) datapoints over a limited temperature  
324 interval of 50-100 °C (e.g. Li & Li, 2011, 2012a). The same observation is made here.  
325 For sample 138101 we have carried out a dose recovery test as a function of prior IR  
326 stimulation temperature (Fig. 6C). It can be seen that dose recovery is satisfactory  
327 over a wide prior IR stimulation temperature interval between 50 and 260 °C for the  
328 pIRIR<sub>290</sub> data. This is in contrast with the MET-pIRIR dose recovery data which shows  
329 a pronounced increase in of dose recovery between 50 and 150 °C IR stimulation  
330 temperature; the shape is very similar to the MET-pIRIR  $D_e$  data shown in Fig. 6A for  
331 the same sample. We conclude that the MET-pIRIR  $D_e$  data are significantly affected  
332 by dose recovery problems at low IR stimulation temperatures (a suggestion to this  
333 effect was made in Li & Li 2011). This is consistent with the observations of Kars *et al.*  
334 (2014b) who showed that high preheats (>300 °C) should not be used with low  
335 temperature IR stimulations. Because of this, we suggest using first IR stimulation

temperature plateaus instead of the MET-method to determine whether a more stable signal has been reached.

**Stability.** Thiel *et al.* (2011a) were the first to show a natural pIRIR<sub>290</sub> signal in saturation on the laboratory dose response curve for a sample collected below the B/M boundary; this led them to conclude that the pIRIR<sub>290</sub> signal is a stable signal. Other studies have made the same observation for pIRIR<sub>290</sub> (Buylaert *et al.* 2011; Thomsen *et al.* 2011) and the high-temperature MET-pIRIR signals (Li & Li 2011, 2012a). In order to investigate this for our material, we collected a sample (code 1535, 27.30 m) below the B/M boundary defined using palaeomagnetic measurements (Zeng *et al.* 2011). The expected burial dose is >2000 Gy and all natural signals are expected to be in field saturation.

Natural signals and dose response curves were measured for both the pIRIR<sub>290</sub> signals (using different prior-IR stimulation temperatures) and the MET-pIRIR signals. The natural signals are shown in Fig. 7, expressed as a fraction of the saturation level of the laboratory dose response curves. Note that for simplicity a single saturating exponential was fitted through the data; Guralnik *et al.* (2015) have shown that for their sample a single exponential fit gives a  $D_0$  indistinguishable from that obtained from their theoretically based generalised growth curve. Because most earlier work

tended to use relatively small test doses, in this experiment we use both a large (500 Gy, approximately equal to  $D_0$ ) and a small (50-60 Gy) test dose.

The results obtained using a 500 Gy test dose are discussed first (Fig. 7A). For the pIRIR<sub>290</sub> protocol, lower first-IR stimulation temperatures (50-140 °C) yield fraction of saturation values of less than 0.90 (average =  $0.87 \pm 0.01$ ) and higher temperatures (170-260 °C) yield higher values (average =  $0.92 \pm 0.01$ ). The MET-pIRIR data measured with a 500 Gy test dose does not seem to show a clear plateau at higher temperature (previously reported at 250-300 °C; see Fig. 3 in Li & Li 2012a). At 250 and 300 °C the fraction of saturation is  $0.93 \pm 0.01$  and  $0.965 \pm 0.004$ , respectively. Both for pIRIR<sub>290</sub> (measured with prior IR stimulation  $\geq 170$  °C) and the MET-pIRIR protocols, the data are not consistent with unity (~4% below saturation light level for MET and ~8% for pIRIR<sub>290</sub>) which might suggest instability in the pIRIR<sub>290</sub> signal and the high-temperature MET-pIRIR signals. For the pIRIR<sub>290</sub> signals measured with low prior-IR stimulation temperatures this underestimation increases to ~13%.

Fig. 7B shows the results when a small test dose of 50-60 Gy is used. For both pIRIR<sub>290</sub> (only measured at 50 and 200°C prior-IR stimulation temperature) and the MET-pIRIR signals the fraction of saturation appears to have increased significantly. For the pIRIR<sub>200,290</sub> this value is  $0.99 \pm 0.02$ , consistent with the MET-pIRIR signals measured at 250 and 300 °C ( $0.96 \pm 0.02$ ); this could be interpreted as suggesting

negligible instability. However, given the test dose dependence of the dose recovery and  $D_e$  results, it is not surprising that the fraction of saturation reached by a natural signal is also a function of test dose size. One must be critical of saturation measurements made with a test dose of only 60 Gy ( $\ll 10\%$  of the natural dose). The measurements made with a 500 Gy test dose ( $\sim D_0$ ) are more likely to yield accurate  $D_e$  measurements and so more accurate measurements of saturation level. We conclude that the apparent stability of signals (e.g. Fig. 1 in Buylaert *et al.* 2012 - pIRIR; Li & Li 2012a – MET-pIRIR) measured with a small test dose is likely to be an artefact of the dose recovery dependence on test dose size, and that the small but finite apparent instability (up to  $\sim 8\%$  below saturation) observed at saturation using a large test dose is probably real.

To further investigate the stability of pIRIR signals we have measured fading rates ( $g_{2\text{days}}$  values) on sample 138106 using a 50 and 200 °C prior-IR stimulation temperature. The resulting  $g_{2\text{days}}$  values are  $0.75 \pm 0.10\%$ /decade ( $n = 6$ ) and  $0.37 \pm 0.13\%$ /decade ( $n = 6$ ) for pIRIR<sub>50,290</sub> and pIRIR<sub>200,290</sub> respectively. It appears that the pIRIR<sub>200,290</sub> fading rate is lower than the pIRIR<sub>50,290</sub> fading rate; this is consistent with the saturation measurements on sample 1535 presented earlier. Li & Li (2012b) have also argued that pIRIR<sub>200,290</sub> is more stable than pIRIR<sub>50,290</sub>.

### ***Bleaching characteristics***

**Comparison of quartz OSL, feldspar IR<sub>50</sub> and pIRIR<sub>290</sub> bleaching rates.** Fig. 8A

summarises the quartz OSL, IR<sub>50</sub> and pIRIR<sub>290</sub> ages for all 15 samples. All the IR<sub>50</sub> data are consistent with a smooth curve passing through the origin and lying below the 1:1 line; this is what would be expected from samples for which both signals are well-bleached at deposition but for which the IR<sub>50</sub> signal is significantly unstable compared to pIRIR<sub>290</sub> (Buylaert *et al.* 2013). This result is not surprising given the prolonged and almost ideal light-exposure received by wind-blown dust before final deposition. Note that this relationship does not arise because of a dose-dependent change in initial sensitivity of the IRSL signals (see Fig. S1). Although the scatter in the quartz and pIRIR age relationship is larger, the data are consistent with the fitted curve and there is again no evidence for pIRIR<sub>290</sub> outliers at larger ages.

To illustrate the likely initial conditions of this material had it not been well-bleached, we exposed quartz and feldspar aliquots of the oldest sample (138115) to a SOL2 spectrum at a lamp-sample distance of ~80 cm for various lengths of time. It took ~130 s for the pIRIR<sub>290</sub> dose, ~20 s for the IR<sub>50</sub> dose and <2 s for the quartz OSL dose to be reduced by 50%. The resulting residual dose are shown as open circles (IR<sub>50</sub>) and open stars (quartz OSL) in Fig. 8B, plotted against the residual pIRIR<sub>290</sub> doses. The differential bleaching rates of the three signals are obvious and each dataset has been fitted with a single and double exponential; when the residual quartz OSL dose is less than 10% of its initial value the IR<sub>50</sub> dose is ~55% of its initial

value and the pIRIR<sub>290</sub> is >80%, comparable to the results described by Murray *et al.* (2012). As expected both curves point at the origin – sufficient light exposure will bleach all three signals so that the apparent residual doses are close to zero. Had our loess samples been incompletely bleached in nature at deposition then the observed IR<sub>50</sub> and pIRIR<sub>290</sub> doses would have grown along a line parallel to the solid line but starting from some point on the dashed line passing through the open circles, depending on the degree of incomplete bleaching. Given the inevitable variation in the degree of incomplete bleaching with time this would have resulted in scattered points lying below the observed solid line (as was seen by Buylaert *et al.* 2013). Since our data do not show such scatter we conclude that the pIRIR<sub>290</sub> signals (and thus the IR<sub>50</sub> and quartz OSL signals) from these samples were all well-bleached at deposition.

**Residual component.** Thomsen *et al.* (2008) and Buylaert *et al.* (2009) have shown that some pIRIR signals can be readily bleached (with residual doses <2 Gy); these levels are insignificant when older sediments are dated. However, more recent work has found a much wider ranges in residual doses (2 to >20Gy) after natural and laboratory bleaching of the pIRIR<sub>290</sub> signal (Thiel *et al.* 2011a, b; Buylaert *et al.* 2011, 2012; Stevens *et al.* 2011; Lowick *et al.* 2012; Murray *et al.* 2012, 2014; Sohbati *et al.* 2012; Kars *et al.* 2014a). The curve fitted through the open circles in Fig. 8B suggests that it is possible to bleach the pIRIR<sub>290</sub> signals to relatively small doses but it is not

440 clear whether there may be an unbleachable component. To investigate the degree to  
 441 which our pIRIR<sub>290</sub> signal is bleachable, we have repeated the experimental  
 442 procedures described by Sohbati *et al.* (2012). A set of 225 aliquots was prepared for  
 443 each of three samples from the top, middle and bottom of the of the loess sequence  
 444 (138102, 138108, 138115). These were exposed in groups of three to twelve aliquots  
 445 per sample for various lengths of time (3.7 to 4171 h) in a Hönle SOL2 simulator at a  
 446 lamp-sample distance of 80 cm. The apparent dose was measured using the pIRIR<sub>290</sub>  
 447 protocol. The results are summarized in Fig. 9A (pIRIR<sub>290</sub>) and Fig. 9C (IR<sub>50</sub>). There is  
 448 a clear tendency for the residual doses to increase with equivalent dose ( $D_e$ ).  
 449 Extrapolation of these residual doses to a  $D_e$  of zero Gy can be used to predict the  
 450 average residual dose that would have been present in these samples before burial  
 451 had they been exposed to these light levels (Buylaert *et al.* 2012; Sohbati *et al.* 2012;  
 452 Kars *et al.* 2014a). The inset to Fig. 9A shows this relationship for all 15 samples for a  
 453 15 h Sol2 bleach. The intercepts of the three samples bleached for various lengths of  
 454 time are plotted against exposure time in Fig. 9B,D for pIRIR<sub>290</sub> and IR<sub>50</sub> signals,  
 455 respectively. In both cases this residual appears to be consistent with a constant (or  
 456 very difficult to bleach) dose after a bleaching time of ~300 h. In our view it is likely  
 457 that these apparently constant difficult-to-bleach signals were present in these  
 458 samples at the time of deposition and accordingly a pIRIR<sub>290</sub> residual dose of  $6.2 \pm 0.7$   
 459 Gy has been subtracted from all measured  $D_e$  values used to calculate the pIRIR<sub>290</sub>  
 460 ages given in Table 1.

## A chronology for the NYZG section: comparison of quartz and feldspar ages

Fig. 10A presents the quartz and pIRIR<sub>290</sub> feldspar ages on a simplified section of the NYZG site. All of the quartz and feldspar ages are consistent with each other down to a depth of 2.60 m corresponding to an age of ~70 ka. The quartz dose in the 71±5 ka sample (138112 at 2.60 m) is 212±10 Gy, consistent with the ~200 Gy limit suggested by Buylaert *et al.* (2007, 2008). This agreement is clearer in Fig. 8A which shows that there is no easily detectable systematic deviation between quartz and feldspar below ~80 ka. Only the oldest three samples show significant deviation.

The data suggest that the clearly defined soil at the bottom of this section below 2.8 m is consistent with deposition during the MIS 5 (130-75 ka). The sedimentation rate during this period increases dramatically from  $0.0063^{+0.0034}_{-0.0017}$  m ka<sup>-1</sup> during the interglacial to  $0.16^{+0.12}_{-0.05}$  m ka<sup>-1</sup> during the subsequent glacial period. The L<sub>1</sub> loess from ~2.7 up to 1.2 m accumulated over a time period that is small compared to our uncertainties. There may be a discontinuity above 1.2 m where the feldspar age drops from 59±3 to 44±3 ka, and another between 0.8 m (43±2 ka) and 0.6 m (32±2 ka). However, the spatial resolution of the data is insufficient to be confident of these breaks and we confine ourselves to calculating the average sedimentation rate for the



top 1 m of  $0.031 \pm 0.005 \text{ m ka}^{-1}$ . The OSL ages confirm that the Holocene soil is missing but the weak soil developed during the warming period of the Last Glacial, i.e. MIS 3, so we cannot tell whether recent deposition rates are comparable with the previous interglacial.

It appears that sedimentation rate was low during MIS 5 and accelerated at the onset of MIS 4. At MIS 4 there was a period of very high dust deposition followed by slow deposition throughout MIS 3.

## Conclusions

We have examined both quartz and feldspar from the NYZG loess deposit in northeastern China in detail. The quartz is sensitive, fast component dominated and not surprisingly has excellent luminescence characteristics

Based on studies of dose recovery and equivalent dose as a function of test dose size measured using the pIRIR<sub>290</sub> signal, we deduce that the test dose employed should be related to the equivalent dose under investigation. Typically it would appear that a test dose of 15 to 80% of the  $D_e$  is most likely to give a satisfactory dose estimate. Furthermore, we question the interpretation of MET-pIRIR data in terms of increasing stability with increasing stimulation temperature. Even in a sample of

non-finite feldspar age, the signal resulting from a low first-IR stimulation temperature in a pIRIR<sub>290</sub> protocol only results in a sensitivity corrected luminescence signal ~5% lower than that resulting from a high first-IR stimulation temperature; contrast this with the ~21% underestimate at low MET-temperatures. It appears that this underestimate is at least in part an artefact of variation in dose recovery with stimulation temperature. Nevertheless, both methods suggest the presence of some instability (~10% signal loss) at field saturation even at the highest (first IR) stimulation temperature (Fig. 7).

Finally, we have investigated the relative bleaching characteristics of quartz OSL and the IR<sub>50</sub> and pIRIR<sub>290</sub> feldspar signals and confirmed that quartz OSL bleaches considerably more rapidly than feldspar, reaching <10% of its initial value when IR<sub>50</sub> has only lost <50% and pIRIR<sub>290</sub> <20%. These data support the suggestion that the ratios of the three signals can be used as indicators of the degree of bleaching of the more sensitive signals. Our pIRIR<sub>290</sub> data are also consistent with an unbleachable residual signal (equivalent to  $6.2 \pm 0.7$  Gy) underlying an exponentially bleached signal.

At this site, quartz OSL and pIRIR<sub>290</sub> ages are in agreement back to ~70 ka. For older sediments we deduce that quartz increasingly underestimates the deposition age. Based on our preferred ages we conclude that sedimentation rates increased rapidly during the Last Glacial, peaking at MIS 4 (~60 ka) at  $0.16^{+0.12}_{-0.05}$  m ka<sup>-1</sup>. During

MIS 3 this sedimentation rate was considerably lower but remained high at  $0.031 \pm 0.005 \text{ m ka}^{-1}$ . Unfortunately, MIS 2 and 1 are missing at this site.

This study has demonstrated the consistency of quartz and feldspar ages (and probably their reliability) over the last glacial period. Beyond that, quartz is increasingly unreliable and it appears that feldspar ages are consistently more accurate.

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710

## 711 **Figure captions**

712

- 713 Fig.1. Map of loess distribution in northeastern China showing location of the study  
 714 site (Niuyangzigou, NYZG) and its stratigraphy. L and S represent loess and  
 715 palaeosol units, respectively. From the top to the bottom, the loess–soil sequences

are named as  $L_1$  and  $S_1$  followed the designation of Liu (1985). Based on the OSL data the Holocene soil ( $S_0$ ) appears to be missing.

Fig.2. Luminescence characteristics for coarse-grained (63-90  $\mu\text{m}$ ) quartz. A. and B. Dose–response curves for aliquots of samples 138101 and 138115 respectively (inset shows the natural decay curves measured at 125 °C and at 90% blue LED power). C. Preheat plateau tests of samples 138101 and 138115. Three aliquots were measured at each temperature and error bars represent 1 standard error. The dash-dot line is drawn at the average  $D_e$  over the 180-280 °C interval. D. Dose recovery ratio as a function of preheat temperature for samples 138101 and 138107. Aliquots were first bleached with blue light at room temperature (2×100 s separated by a 10 ks pause) and then given a dose of 78.5 Gy and 156 Gy for 138101 and 138107, respectively. The solid line is drawn at unity and the dashed lines at  $\pm 10\%$ . Three aliquots were measured per preheat temperature and error bars represent one standard error. The inset shows a histogram of recycling ratios for all samples (measured as part of  $D_e$  determination).

Fig.3. A. Natural signals and dose–response curves for coarse-grained quartz and K-feldspar of sample 138115. Data were fitted with a sum of two saturating exponential functions of the form  $y = ax(1 - \exp(-bx)) + cx(1 - \exp(-dx))$ . The sensitivity-corrected luminescence ( $L_x/T_x$  values) was normalized to the fitted

saturation values ('a+c'). B.  $L_x/T_x$  plots for the pIRIR<sub>290</sub> signal for a set of samples down the section. Three aliquots were measured for each sample but only one representative aliquot is shown. Sample 138101 ( $L_x = 100$  Gy;  $T_x = 30$  Gy); 138102 ( $L_x = 999$  Gy;  $T_x = 300$  Gy); 138108 ( $L_x = 216$  Gy;  $T_x = 65$  Gy); 138115 ( $L_x = 362$  Gy;  $T_x = 108$  Gy) and the B/M boundary sample 1535 ( $L_x = 999$  Gy;  $T_x = 300$  Gy).

Fig.4. Result of the dose recovery test for pIRIR<sub>290</sub>. A. Plot of the dose recovery ratio versus test dose size. The test dose size varies from 5 to 260% of the total (natural+added) dose. The dose recovery ratio was calculated as the measured dose divided by the sum of the natural and the given dose. The solid line is drawn at unity and the dashed-dot lines at  $\pm 5\%$ . B. dose recovery results show as the measured dose versus added dose.

Fig.5. Dependence of  $D_e$  on test dose size for the uppermost (138101), middle (138108) and lowermost (138115) samples from the upper 3.2 m section. The dash-dot line is the average of the  $D_e$  value for test doses ranging between 15 and 80% of the total (natural + added) dose.

Fig.6. Comparison of pIRIR<sub>290</sub> and MET-pIRIR. A. and B. Dependence of  $D_e$  on prior IR stimulation temperature for the uppermost (138101) and lowermost (138115) samples from the upper 3.2m section. Three aliquots were measured at each

temperature and error bars represent one standard error. The dash-dot line is drawn at the average  $D_e$  over the 50-260 °C interval for the pIRIR<sub>290</sub>. C. Dependence of dose recovery ratio on prior IR stimulation temperature for the uppermost sample (138101). Three aliquots were measured at each temperature and error bars represent one standard error. The dash-dot line is drawn at unity on the vertical axis.

Fig.7. Fraction of saturation for both the pIRIR<sub>290</sub> and MET-pIRIR signals for a sample from below to the B/M boundary (burial dose >2000 Gy). A. Fraction of saturation measured using the large test dose. B. Fraction of saturation measured using the small test dose. All the data were fitted with a single saturating exponential function. Each data point is an average of three aliquots and error bars represent one standard error. The solid line is drawn at unity and the dashed line at 0.9.

Fig.8. Comparison of age and bleaching rate for the quartz OSL, feldspar IR<sub>50</sub> and pIRIR<sub>290</sub> signals. A. Quartz OSL and feldspar IR<sub>50</sub> ages plotted as a function of pIRIR<sub>290</sub> age. The IR<sub>50</sub> and pIRIR<sub>290</sub> ages have a residual dose of  $0.6 \pm 0.1$  Gy and  $6.2 \pm 0.7$  Gy subtracted from the  $D_e$  and are not corrected for fading. The vertical dashed line shows the upper quartz OSL dating limit. B. Residual quartz OSL, feldspar IR<sub>50</sub> and pIRIR<sub>290</sub> doses for different Hönle SOL2 solar simulator bleaching times for sample 138115. The solid line was derived from fitting the IR<sub>50</sub> to pIRIR<sub>290</sub>  $D_e$  values for all 15 samples. Each data point is an average of three aliquots and error

bars represent one standard error.

Fig.9. The relationship between the residual dose and  $D_e$  obtained with the pIRIR<sub>290</sub> protocol on three samples (138102,-08,-15). A. and C. Average residual dose obtained after different Hönle SOL2 solar simulator bleaching times as a function of the pIRIR<sub>290</sub> and IR<sub>50</sub>  $D_e$ , respectively. Each point is the average residual dose of three to twelve aliquots per sample obtained after the given exposure times and error bars represent 1 standard error. The relationship between residual dose and equivalent dose is described by a linear fit for each exposure time. Inset in A. is the same relationship for all 15 samples for a single bleaching time of 15 h. B. and D. Intercept of the linear fits with the y-axis from A. and C. as a function of exposure time.

Fig.10. Stratigraphy, quartz OSL and feldspar pIRIR<sub>290</sub> ages for NYZG. The ages are plotted against the section depth. Open circles represent the quartz OSL ages and closed circles represent the pIRIR<sub>290</sub> ages. Sedimentation rates were derived from linear regression.

## Table captions

Table 1. Summary of sample code and depth, radionuclide concentrations, calculated

800 dose rates, OSL and pIRIR<sub>290</sub> D<sub>e</sub> values and luminescence ages. The absolute  
801 uncertainty on the water content is  $\pm 5\%$ . The pIRIR<sub>290</sub> D<sub>e</sub> values have a residual dose  
802 of  $6.2 \pm 0.7$  Gy subtracted from the measured value while the pIRIR<sub>290</sub> ages are not  
803 corrected for fading. (n) denotes the number of aliquots contributing to the D<sub>e</sub>.

804

805 Table 2. Outline of dose measurement protocols used in this study. SAR protocol  
806 after Murray & Wintle (2000, 2003), post-IR IRSL protocol after Thiel *et al.* (2011a),  
807 MET-post-IR IRSL after Li & Li (2012a). For the 'natural' sample, the give dose = 0. T  
808 varies from 50 to 260 °C. The whole sequence is repeated for several regenerative  
809 doses including a zero dose and a repeat dose.